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14. ABSTRACT
The goal of the project is to develop robust sensing methodologies based on carbon nanotube for the detection of damage in advanced army composite systems. The fundamental idea is that carbon nanotubes can function as a network of sensors in traditional laminated fiber composites to detect deformation and damage in situ, using electrical techniques.

The novelty of the approach is in the use of carbon nanotube based multifunctional composites which integrate

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Report Title

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The goal of the project is to develop robust sensing methodologies based on carbon nanotube for the detection of damage in advanced army composite systems. The fundamental idea is that carbon nanotubes can function as a network of sensors in traditional laminated fiber composites to detect deformation and damage in situ, using electrical techniques.

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The innovative features of the project include: (a) The processing of SC-15 epoxy based glass fiber composites with highly uniform dispersion of carbon nanotubes as electrical conductive network; (b) The characterization of damage sensing capability of the hybrid composite under static and dynamic loading, and (c) The capability in analysis/modeling of 3D electrical percolation behavior and the evolution of damage with deformation.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received		Paper
08/03/2011	2.00	Limin Gao, Tsu-Wei Chou, Erik T. Thostenson, Zuoguang Zhang, Magali Coulaud. In situ sensing of impact damage in epoxy/glass fiber composites using percolating carbon nanotube networks, Carbon, (8 2011): 0. doi: 10.1016/j.carbon.2011.04.003
08/03/2011	3.00	Weibang Lu, Tsu-Wei Chou. Analysis of the entanglements in carbon nanotube fibers using a self-folded nanotube model, Journal of the Mechanics and Physics of Solids, (3 2011): 0. doi: 10.1016/j.jmps.2011.01.004
08/03/2011	4.00	Weibang Lu, Tsu-Wei Chou, Byung-Sun Kim. Radial deformation and its related energy variations of single-walled carbon nanotubes, Physical Review B, (4 2011): 134113. doi: 10.1103/PhysRevB.83.134113
08/09/2012	5.00	Amanda S. Wu, Tsu-Wei Chou, John W. Gillespie, David Lashmore, Jeff Rioux. Electromechanical response and failure behaviour of aerogel-spun carbon nanotube fibres under tensile loading, Journal of Materials Chemistry, (02 2012): 0. doi: 10.1039/c2jm15869h
08/10/2012	6.00	Amanda S. Lim, Qi An, Tsu-Wei Chou, Erik T. Thostenson. Mechanical and electrical response of carbon nanotube-based fabric composites to Hopkinson bar loading, Composites Science and Technology, (03 2011): 0. doi: 10.1016/j.compscitech.2010.12.025
08/16/2013	7.00	Tsu-Wei Chou, Limin Gao, Erik T. Thostenson, Zuoguang Zhang, Joon-Hyung Byun. An assessment of the science and technology of carbon nanotube-based fibers and composites, Composites Science and Technology, (1 2010): 0. doi: 10.1016/j.compscitech.2009.10.004
08/16/2013	8.00	Tsu-Wei Chou, Erik T. Thostenson, Ajay Godara, Zuoguang Zhang, Luca Mezzo, Limin Gao. Highly conductive polymer composites based on controlled agglomeration of carbon nanotubes, Carbon, (8 2010): 0. doi: 10.1016/j.carbon.2010.03.027
08/16/2013	9.00	Weibang Lu, Tsu-Wei Chou, Erik T. Thostenson. A three-dimensional model of electrical percolation thresholds in carbon nanotube-based composites, Applied Physics Letters, (2010): 0. doi: 10.1063/1.3443731
08/19/2013	10.00	Amanda S. Wu, Tsu-Wei Chou. Carbon nanotube fibers for advanced composites, Materials Today, (7 2012): 0. doi: 10.1016/S1369-7021(12)70135-9
TOTAL:		**9**

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received	Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Research results have been presented at the following conferences:

2009

1. 50th AIAA/ASME/ASCE/AHS/ASC SDM Conference, Palm Springs, CA
2. 17th International Conference on Composites Materials, Edinburgh, UK (Plenary Lecture)
3. 1st Joint Canadian and American Composites Society Technical Conference, Newark, DE
4. 4th International Conference on Carbon Based Nanocomposites, Hamburg-Harburg, Germany
5. FRP Composites for Infrastructure Applications, San Francisco, CA

2010

1. TMS Annual Meeting and Exhibition, Seattle, Washington (Keynote Lecture)
2. Materials and Surface Science Institute, University of Limerick, Ireland (Distinguished Lecture Series)
3. 1st TMS-ABM International Materials Congress, Rio de Janeiro, Brazil (Invited Speaker)
4. ASC 25th Annual Technical Conference, 14th US-Japan Conference on Composite Materials, Dayton, Ohio
5. 2nd International Symposium on Nanomechanics and Nanocomposites, Beijing, China (Keynote Lecture)
6. School of Aerospace Engineering, Beijing Institute of Technology, Beijing, China (Invited Speaker)
7. Tianjin University, Tianjin, China (Invited Speaker)
8. 7th Asian-Australasian Conference on Composite Materials, Taipei, Taiwan (Keynote Lecture)
9. MRS Annual Conference, Kaoshong, Taiwan (Keynote Lecture)

2011

1. The 2nd Symposium for the Global Research Laboratory (GRL), Seoul, Korea (Invited Speaker)
2. Aerospace Applications of Nanotechnology Lecture Series, Boeing Comp. (two WebEx Presentations)
3. Deformation and Fracture of Composites (DFC-11), Cambridge, UK
4. The 4th World Materials Research Institutes Forum, Shenyang, China (Invited Speaker)
5. The 5th Asia-Europe Symposium on Processing and Properties of Reinforced Polymers, Dresden, Germany (Keynote Speaker)
6. The 14th International Conference on Advances in Materials and Processing Technology (AMPT2011), Istanbul, Turkey
7. The 11th U.S. National Congress on Computational Mechanics (USNCCM11), Minneapolis, Minnesota, USA
8. The 18th International Conference on Composite Materials (ICCM18), Jeju Island, Korea (Keynote Speaker)
9. The 26th ASC Annual Technical Conference/ The 2nd Joint US-Canada Conference on Composites, Montreal, Canada
10. ISWHM – Stanford University
11. Army Research Lab., Aberdeen Proving Ground, MD

2012

1. Tongji University, Shanghai, China
2. Zhejiang University, Hangzhou, China
3. Suzhou Institute of Nanotechnology and Nanobionics, Suzhou, China
4. Mechanics of Nano, Micro, and Macro Composite Structures, Politecnico di Torino, Italy (Plenary Speaker)
5. The 2nd International Conference on Advanced Polymer Matrix Composites, Harbin, China (Plenary Speaker)
6. The Third International Symposium on Materials for Enabling Nanodevices, Symposium Honoring Professor James C. M. Li, UCLA (Plenary Speaker)
7. The 17th National Conference on Composite Materials, Beijing, China (Plenary Speaker)

2013

1. Korea Institute of Materials Science, Changwon, Korea
2. Seoul national University, Seoul, Korea
3. Florida International University, Miami, FL
4. Tongji University, Shanghai, China
5. DFC12 and SI6, Kelly Symposium, Cambridge, UK
6. 6th Asia-Europe Symposium on Processing and Properties of Reinforced Polymers, Wuhan, China (Keynote Lecture)
7. ICCM-19, Montreal, Canada (Keynote Lecture)
8. Leuven, Belgium (Keynote Lecture)

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received		Paper
08/03/2011	1.00	Amanda S. Lim, Qi An, Tsu-Wei Chou, Erik T. Thostenson. Mechanical and electrical response of carbon nanotube-based fabric composites to Hopkinson bar loading, Composites Science and Technology (03 2011)
TOTAL:		**1**

Number of Manuscripts:

Books

Received Paper

TOTAL:

Patents Submitted

Patents Awarded

Awards

2013:
Advisory Professor, Tongji University, Shanghai, China
Nadai Medal, ASME
2011:
Top 100 Materials Scientists (Ranked #34), Times Higher Education
World Fellow, International Committee on Composite Materials
2009:
Medal of Excellence in Composite Materials, University of Delaware Center for Composite Materials

Graduate Students

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period:	0.00
The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:......	0.00
The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:......	0.00
Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):	0.00
Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:	0.00
The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense	0.00
The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:	0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED

FTE Equivalent:

Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See Attachment

Technology Transfer

Advanced Composite Armor: *In Situ* Sensing with Carbon Nanotube Networks for Improved Damage Tolerance

Tsu-Wei Chou (PI), W911NF-09-1-0114

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126 Spencer Laboratory, Newark, DE 19716-3140

Table of Contents

1. Statement of the problem studied

The novelty of the approach is in the use of carbon nanotube based multifunctional composites which integrate self-sensing function to structural components and can monitor damage initiation and propagation, and elucidate the nature and extent of damage. The objectives have been accomplished through integrated and complementary analytical and experimental studies.

The innovative features of the project include: (a) The processing of SC-15 epoxy based glass fiber composites with highly uniform dispersion of carbon nanotubes as electrical conductive network; (b) The characterization of damage sensing capability of the hybrid composite under static and dynamic loading, and (c) The capability in analysis/modeling of 3D electrical percolation behavior and the evolution of damage with deformation.

2. Significance

The significance of the research is in the development of technologically advanced materials for armor protection with improved damage tolerance and lower areal density.

The materials typically used in composite armor backing plates are composed of 2-D and 3-D textile fabrics infused with a toughened epoxy matrix. These materials pose fundamental challenges for *in situ* sensing. First, the toughened epoxies may undergo phase separation and produce a barrier toward the development of a percolating network of carbon nanotubes. In addition, the textile structure, whether it is a 2-D plain weave fabric or a 3-D orthogonal weave, adds microstructural complexity which will have a substantial impact on the measured resistance change.

Eventual defense and/or industrial usage may be found in multifunctional applications including advanced composite armor, electromagnetic shielding and electrostatic discharge.

3. Summary of the most important results

3.1 Novel Approach of Carbon Nanotube Network Formation and Their Sensing of Damage [1,2,3]

Carbon nanotube/SC-15 epoxy composites were processed using both calendering and fiber sizing approaches. In the calendering method, a three-roll mill was used to disperse nanotubes in the resin prior to infusion. Glass fibers were treated with a fiber sizing agent containing carbon nanotubes using VARTM before resin infusion. We found that both methods achieved electrical percolation however the fiber sizing agent was able to better distribute the nanotubes within the thick-section fabric preforms due to its significantly lower viscosity. A microstructural analysis of these carbon nanotube/UD S-2 glass fiber/SC-15 epoxy composites has been performed and the result has been published (Fig. 1).

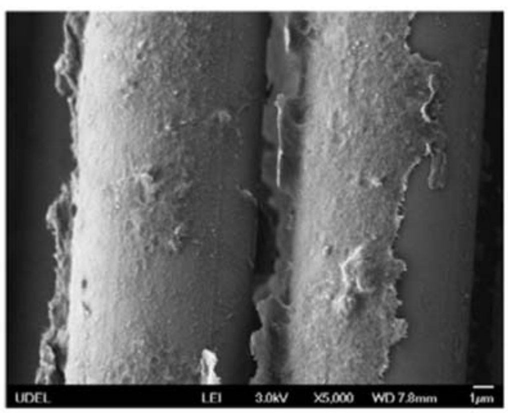


Fig. 1 Carbon nanotube deposited on the surface of glass fibers using a carbon nanotube sizing agent

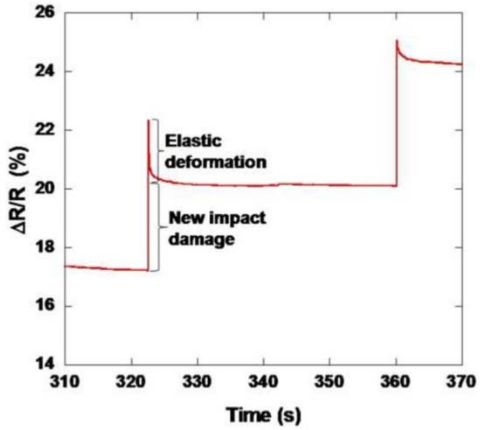


Fig. 2a. Resistance increase during impact shows elastic deformation and new impact damage

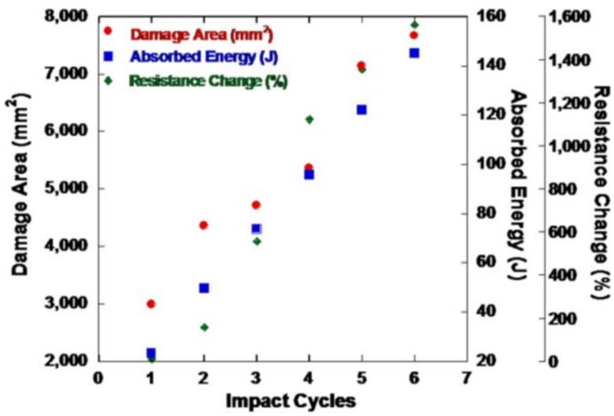


Fig. 2b. Comparative study of resistance change and damage area (measured by ultrasonic C-scan) and absorbed energy

Research into the dynamic behavior of these composites was performed using an Instron Datup 8250 drop-weight impact testing machine. During these experiments, specimens were impacted multiple times with energies of 70 J. After each successive impact, resistance increases are related with damage area and absorbed energy (Fig. 2a and 2b).

3.2 Modeling the Electrical Percolation Threshold of the Carbon Nanotube Network [4]

A three-dimensional model of carbon nanotube networks was developed and implemented to evaluate the role of intertube van der Waals interactions and electrical tunneling on electrical percolation (Fig. 3a and 3b).

Through this study, we reveal that van der Waals interactions and tunneling play a significant role in the electrical percolation threshold of a network of low aspect ratio carbon nanotubes (Fig. 4a). In the case of a high aspect ratio network, these two interactions become less significant. Carbon nanotube waviness is also shown to have a strong effect on electrical percolation; the threshold increases gradually with the maximum nanotube deviation angle (Fig. 4b). Through these simulations, a better understanding of percolation in nanotube networks is obtained and processing methods can be adjusted in order to achieve electrical percolation with even lower nanotube volume fractions.

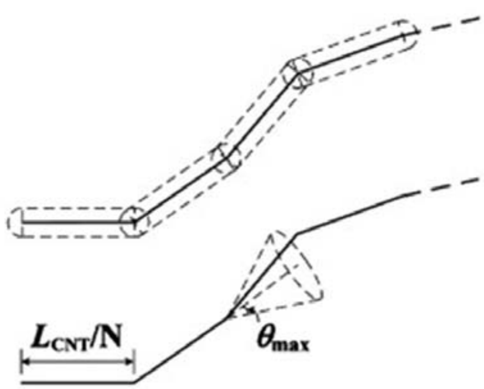


Fig. 3a. A wavy CNT with deviation angle θ_{max}

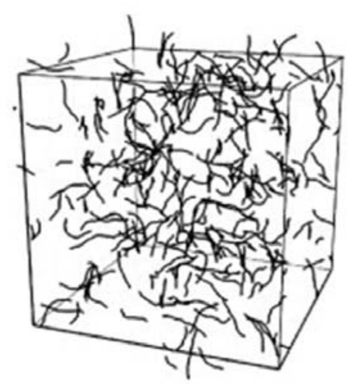
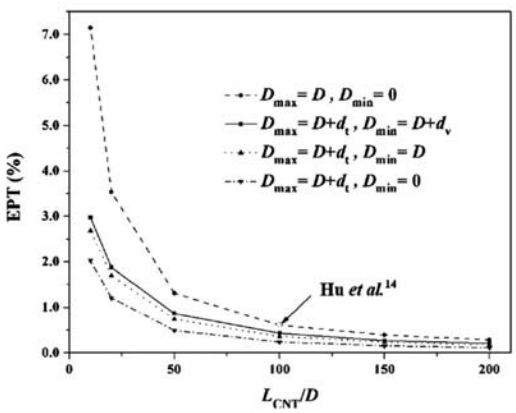


Fig. 3b. A 3D simulation cell of wavy CNTs with N=10 (per CNT) and θ_{max}= 30°.



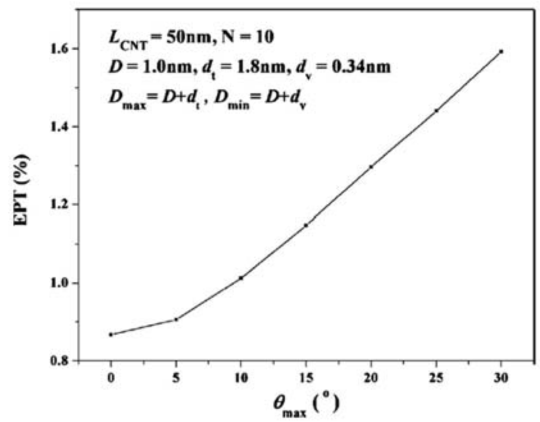


Fig. 4a. Electrical percolation threshold of nanocomposites with straight CNTs vs. CNT aspect ratio

Fig. 4b. The effect of maximum nanotube deviation angle on nanocomposite percolation thresholds.

3.3 Dynamic Behavior - Split Hopkinson Pressure Bar (SHPB) Loading Response [5]

Carbon nanotube/SC-15 epoxy composites were processed using a carbon nanotube fiber sizing agent prior to resin infusion. Both the sizing agent and epoxy resin were applied using VARTM methodology. In order to evaluate the response of thick-section composites (0.35 in/8.9 mm) under dynamic compression loading, 45º off-axis specimens were prepared (Fig. 5).

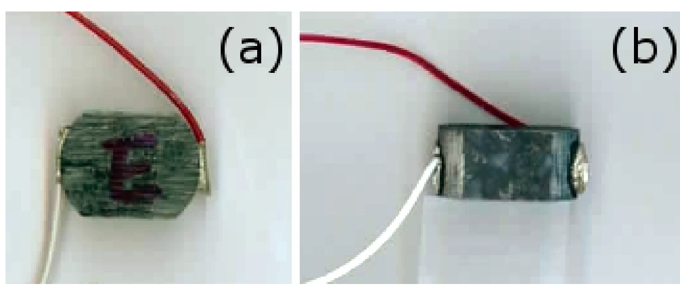
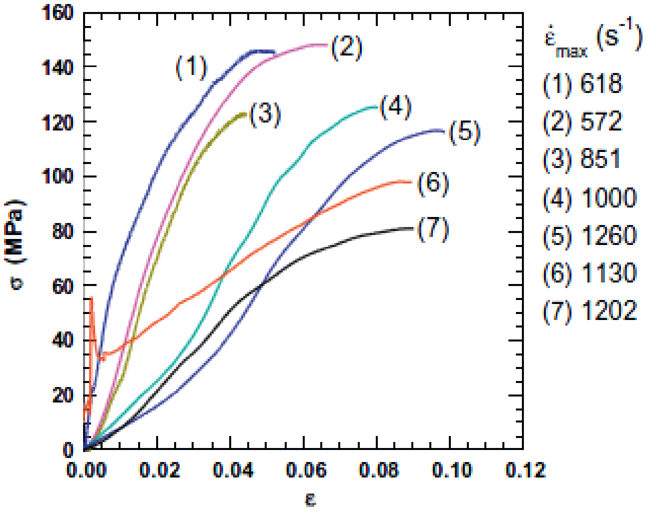


Fig. 5. Composite specimens with electrodes (white and red wires) used in SHPB evaluation

A single specimen was impacted multiple times at increasing impact energy (analogous to striker bar impact velocity). Evidence of progressive damage is seen in the mechanical (Fig. 6a) and electrical (Fig. 6b) response of the composite. Large-scale delamination occurs during the sixth impact; the result is a further decline in stiffness and drastic increase in electrical resistance.



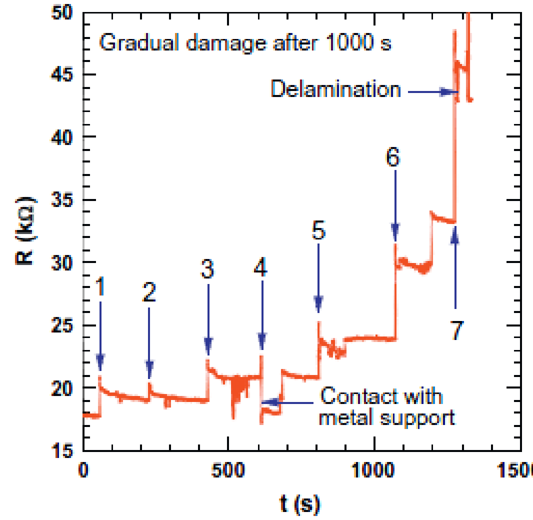
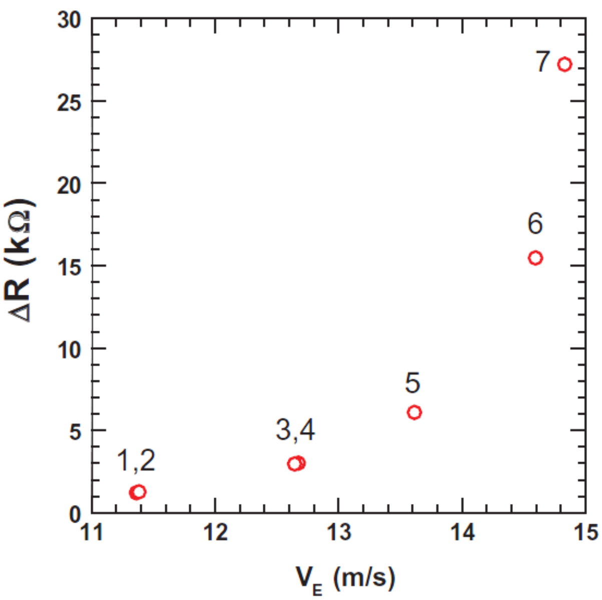


Fig. 6a. Mechanical response to multiple impacts for a single specimen. Specimen stiffness decreases after each successive impact, beginning with the third.

Fig. 6b. Electrical resistance (acquired at 15 Hz) shown throughout loading sequence. Increases in unloaded resistance after impact are indicative of microscale damage development.



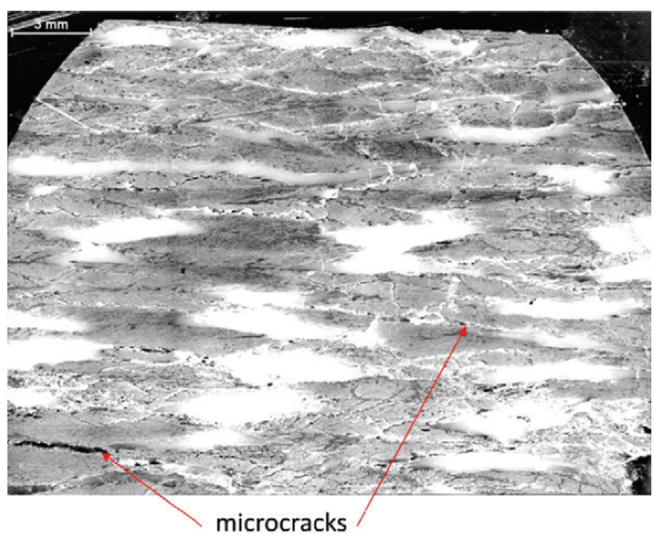


Fig. 7a. Change in baseline resistance vs. striker bar impact velocity, V_E. Numbers indicate impact sequence.

Fig. 7b. Optical micrograph of the impacted surface showing cracks throughout the composite matrix.

Resistance increases exponentially with striker bar impact velocity (Fig. 7a), indicating that damage has occurred progressively as impact energy increases. Evidence of damage, which occurs in the form of microcracking and delamination is shown in Fig. 7b.

Current efforts into characterizing the dynamic response of carbon nanotube-based E-glass/epoxy composites include acquiring electrical data in real-time (5 MHz). This will allow for the correlation of stress-strain response during loading (which occurs in <300 μs) with electrical resistance changes, providing more insight into the development of damage during Hopkinson bar loading.

The mechanical and electrical behavior of a specimen loaded within its linear elastic regime is plotted in Fig. 8. Resistance increases during loading due to Poisson-like radial expansion, caused by the applied axial compression. Since electrical resistance is measured across the specimen diameter, resistance increases during loading. This increase is temporary since the specimen is not damaged permanently.

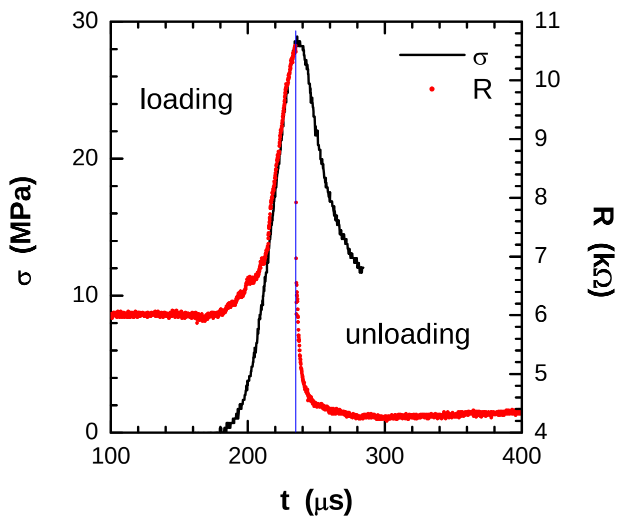


Fig. 8. Stress and electrical resistance (both acquired at 5 MHz) plotted vs. time during a single SHPB loading.

3.4 Analysis and Modeling of Carbon Nanotubes [6, 7]

Entanglements in carbon nanotube fibers and play a crucial role in affecting their mechanical properties. In this study, the carbon nanotube entanglement is modeled as two connecting, self-folded carbon nanotubes. At large aspect ratios, it is energetically favorable for a nanotube to be self-folded due to the van der Waals interactions between different parts of the nanotube. The geometrical characteristics of self-folded carbon nanotubes (SFCNTs), such as the critical length for self-folding as well as the critical effective width and length, are investigated by using both an exact theoretical model and an approximate theoretical model (Fig. 9). The tensile properties of the SFCNTs have been examined by using both the approximate theoretical model and atomistic simulations. Good agreement is observed in the results of these two approaches (Fig. 10).

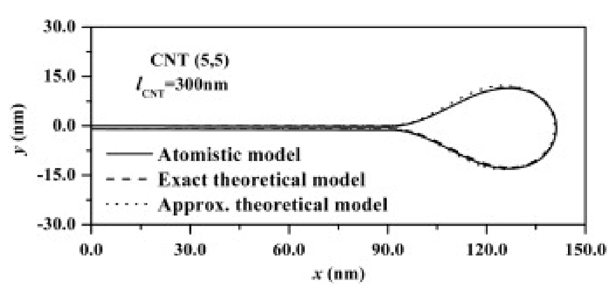


Fig. 9a. Schematic diagram of a SFCNT (5,5) with a length of 300 nm, obtained using atomistic simulations and two theoretical models.

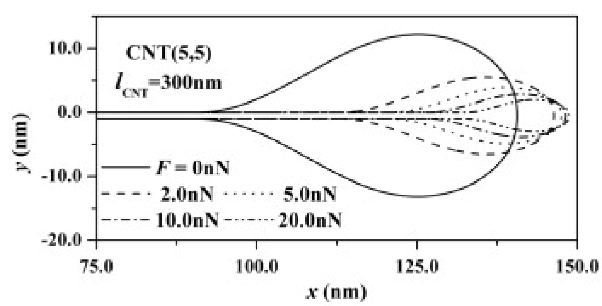


Fig. 9b. Tension-induced deformation of a SFCNT (5,5).

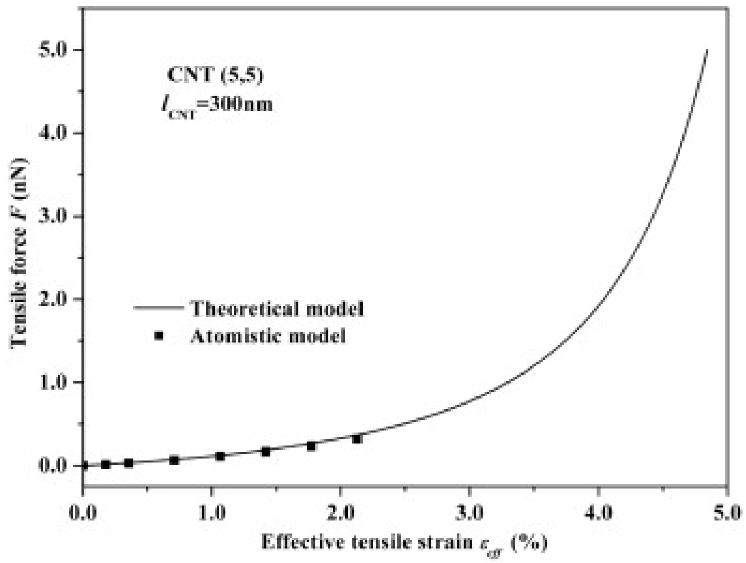


Fig. 10. Predicted load-strain behavior of a SFCNT (5,5) obtained via approximate theoretical analysis and atomistic simulation.

The radial deformation (Fig. 11) of a carbon nanotube plays a significant role in affecting its mechanical and electrical behavior. In this study, both atomistic simulations and continuum analysis are adopted to study the structural transformations and their related energy variations during the radial deformation of single-walled CNTs (SWCNTs).

It has been found that for SWCNTs with radius larger than 1.05 nm, they would collapse under radial deformations. The larger the SWCNT radius, the easier it will collapse. For SWCNTs with radius larger than 1.90 nm, the collapsed states are more stable than their initial, undeformed states. These different behaviors are due to the variation of contributions from the bending strain energy and the van der Waals interaction energy between opposite walls of the SWCNT to the total energy. The energy barrier for the collapse of SWCNTs decreases with increasing SWCNT radius (Fig. 12a), and their relationship is represented by a simple formula. The relationship between the energy difference (the energy variation between the collapsed state and the initial circular state) and SWCNT radius is also obtained (Fig. 12b) and represented by a simple expression.

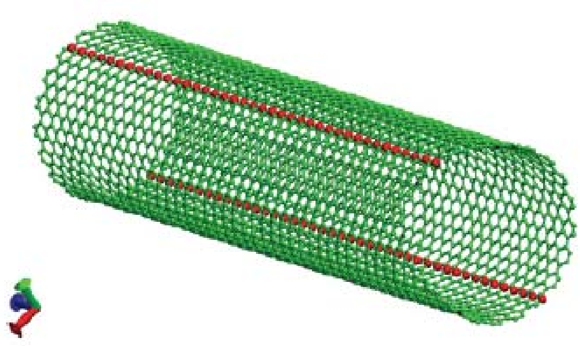


Fig. 11a. Schematic of the initial atomic structure of a carbon nanotube (20,20) with a length of 10.2 nm. Red atoms are forced inward during radial deformation.

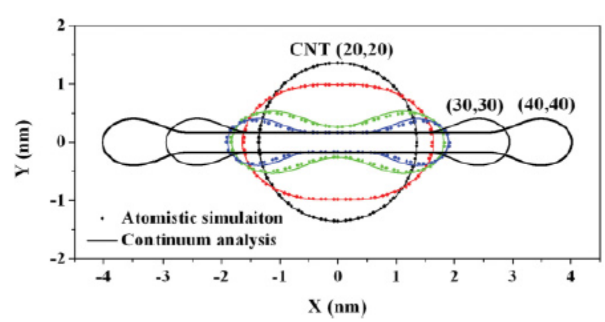
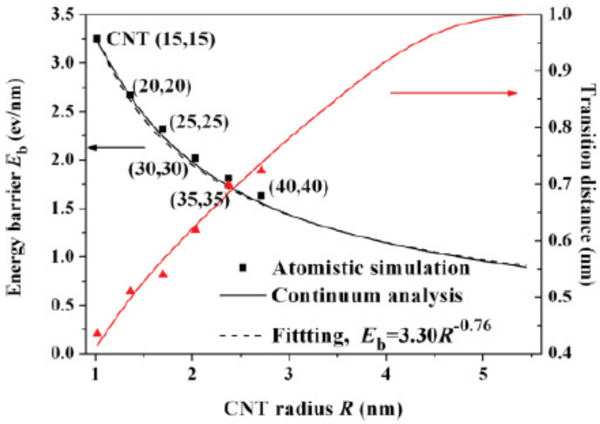


Fig. 11b. Radial deformation sequence of a (20,20) carbon nanotube shown along with collapsed (30,30) and (40,40) carbon nanotubes.



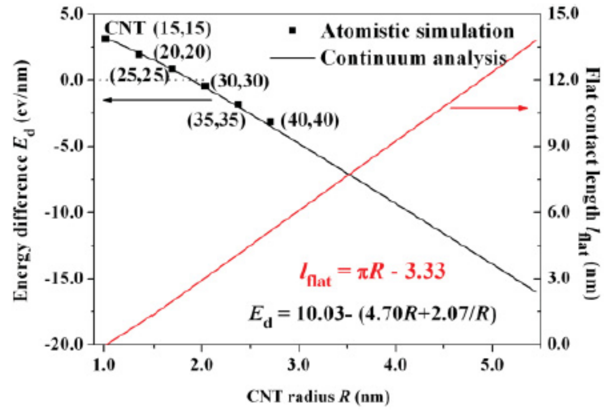


Fig. 12a. Energy barrier and transition distance of different carbon nanotubes under radial deformation. Good agreement is seen between atomistic simulation and continuum analysis.

Fig. 12b. Energy difference and flat contact length of carbon nanotubes with different radii. Again, a strong correlation between the atomistic simulation and continuum analysis is observed.

3.5 Carbon Nanotube Fiber: Electromechanical Response and Failure Behavior [8, 9]

The electromechanical behavior of carbon nanotube fibers (Fig. 13), provided by Nanocomp Technologies, Inc., were evaluated under quasi-static tensile loading. These fibers consisted of single walled carbon nanotubes spun directly from an aerogel state and held together by entanglements and van der Waals interactions. The electrical and mechanical behavior of these fibers is provided in Table 1.

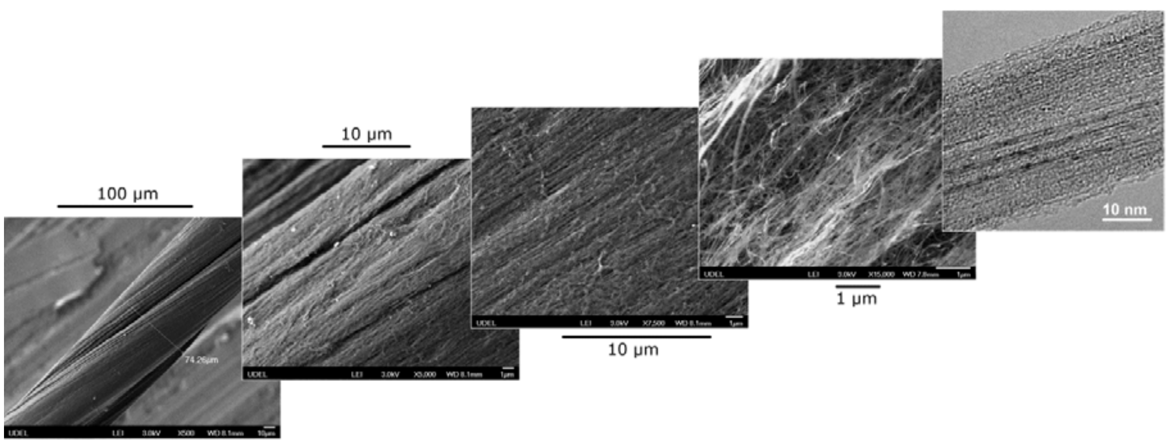


Fig. 13. Composite specimens with electrodes (white and red wires) used in SHPB evaluation

Diameter, d_{avg} [µm]	57.3 ± 5.9
Failure Load, F_{max} [N]	0.49 ± 0.17
Failure Strain, ε_{max}	0.096 ± 0.021
Strength, σ_{UTS} [GPa]	0.189 ± 0.052[a]
Modulus, E [GPa]	9.16 ± 2.53[a]
Conductivity, κ [S m^{-1}]	5.1 ± 1.7 (10^4)
Resistance, R [Ω]	67.5 ± 23.6

Table 1. Material properties of the aerogel-spun carbon nanotube fibers.

The fibers exhibited piezoresistivity upon tensile loading, meaning that they can respond electrically to applied strain and failure. This research provides the foundation for embedding carbon nanotube fibers in composites - the fibers will deform with the composite fibers/matrix and will therefore experience measurable changes in electrical resistance consistent with applied strain. We hypothesize that, as the fibres undergo tensile strain, radial contact between individual carbon nanotubes may improve; however, the degree of axial nanotube-nanotube contact will decrease. This behavior gives rise to increases in electrical resistance observed during loading in Fig. 14.

Upon failure, the carbon nanotubes slide past each other, resulting in lower fiber strengths than the tensile strength of individual carbon nanotubes (Fig. 15). After failure, stress is reflected back through the fiber ends from the point of breakage as a compressive wave. We observed kinking to occur along the fiber length after failure in many cases, leading us to conclude that the compressive strength of the fiber is lower than the tensile strength.

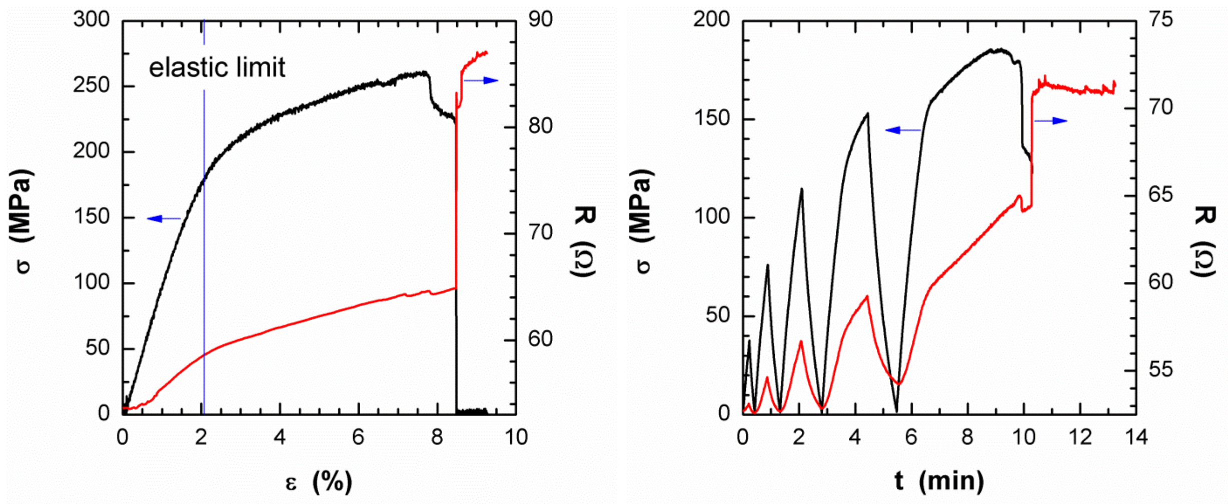


Fig. 14. Typical resistance/stress-strain behavior during monotonic (left) and cyclic (right) tensile loading.

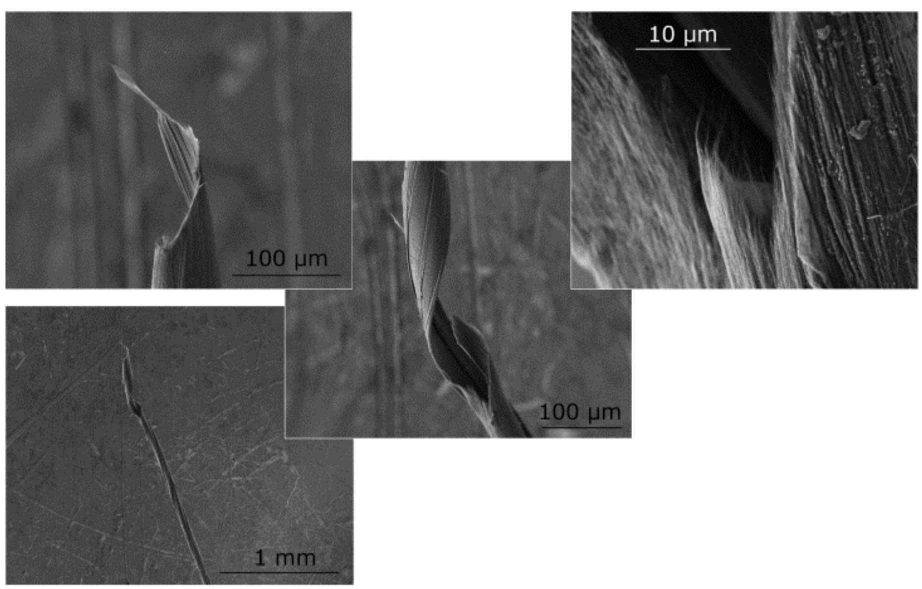


Fig. 15. Failed end of the specimen.

Following the analysis presented by Allen [10] in which it is assumed that the compressive wave is equal in magnitude to the tensile stress applied at failure, it was possible to identify a compressive strength in the range of 172-177 MPa. This behavior was confirmed through loop testing as well.

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5. Technology Transfer

None.

6. Journal Articles

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7. Conference Papers

[1] Chou TW. Recent advancements in the science and technology of carbon nanotube fibers and composites. *2nd International Conference on Advanced Polymer Matrix Composites*. Harbin, China, July 22-25, 2012.

[2] Chou TW. Electromechanical behavior of carbon nanotube fibers and their reinforced composites. *International Conference on Mechanics of Nano, Micro and Macro Composite Structures.* Torino, Italy, June 18-20, 2012.

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8. Awards and Honor of Tsu-Wei Chou

2013:

Advisory Professor, Tongji University, Shanghai, China

Nadai Medal, ASME

2011:

Top 100 Materials Scientists (Ranked #34), Times Higher Education

World Fellow, International Committee on Composite Materials

2009:

Medal of Excellence in Composite Materials, University of Delaware Center for Composite Materials

9. Graduate Students Involved Directly in ARO Project

None.